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14. ABSTRACT The research objective of this project at Alabama A&M University (AAMU), a leading HBCU, was to use nanoengineering and nanofabrication to develop nanostructured thermoelectric (TE) materials for application in high-efficiency thermoelectric power generators and solid-state micro cooling devices. Multi-nano-layered superlattice thin films based on Bi <sub>2</sub> Te <sub>3</sub> /Sb <sub>2</sub> Te <sub>3</sub> , SiO <sub>2</sub> /SiO <sub>2</sub> +CoSb, SiO <sub>2</sub> /SiO <sub>2</sub> +Au, Zn <sub>4</sub> Sb <sub>3</sub> /CeFe(4-x)CoSb <sub>12</sub> , SiO <sub>2</sub> /SiO <sub>2</sub> +Ge, SiO <sub>2</sub> /SiO <sub>2</sub> +Cu, AgBiTe, AgSbTe, Si/Si+Sb, Si/Si+Ge, Zn <sub>4</sub> Sb <sub>4</sub> , and ZrNiSn were grown using ultra-high vacuum based deposition methods. These thin films were further modified by MeV Si ion beam.					
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a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU			Satilmis Budak
					19b. TELEPHONE NUMBER 256-372-5894



## Report Title

Final Report: Development of Thermoelectric Power Generation and Peltier Cooling Properties of Materials for Thermoelectric Cryocooling Devices

### ABSTRACT

The research objective of this project at Alabama A&M University (AAMU), a leading HBCU, was to use nanoengineering and nanofabrication to develop nanostructured thermoelectric (TE) materials for application in high-efficiency thermoelectric power generators and solid-state micro cooling devices. Multi-nano-layered superlattice thin films based on Bi<sub>2</sub>Te<sub>3</sub>/Sb<sub>2</sub>Te<sub>3</sub>, SiO<sub>2</sub>/SiO<sub>2</sub>+CoSb, SiO<sub>2</sub>/SiO<sub>2</sub>+Au, Zn<sub>4</sub>Sb<sub>3</sub>/CeFe(4-x)CoSb<sub>12</sub>, SiO<sub>2</sub>/SiO<sub>2</sub>+Ge, SiO<sub>2</sub>/SiO<sub>2</sub>+Cu, AgBiTe, AgSbTe, Si/Si+Sb, Si/Si+Ge, Zn<sub>4</sub>Sb<sub>4</sub>, and ZrNiSn were grown using ultra-high vacuum-based deposition methods. These thin films were further modified by MeV Si-ion beam bombardment to produce nanodots and nanoclusters for achieving high thermoelectric figure of merit (ZT). To the best of our knowledge, this research team is the only one using high-energy ion-beam technology to enhance the thermoelectric figure of merit. Excellent progress was made to significantly increase the ZT value by increasing the Seebeck coefficient and electrical conductivity while decreasing thermal conductivity for nano-power thermoelectric generators and thermoelectric coolers for achieving cryogenic temperatures. Arguably, Alabama A&M University currently has achieved the highest published room-temperature value of ZT, viz., 4.9, and successfully achieve the research objective. Numerous peer-reviewed papers were published and many presentations were made at conferences. A large number of students participated in this research project.

**Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:**

**(a) Papers published in peer-reviewed journals (N/A for none)**

<u>Received</u>	<u>Paper</u>
04/28/2015 31.00	S. Budak, S. Guner, C. Muntele, D. Ila. Thermoelectric Generators from AgBiTe and AgSbTe Thin Films Modified by High-Energy Beam, Journal of Electronic Materials, (01 2015): 0. doi: 10.1007/s11664-014-3581-8
04/28/2015 35.00	E. Gulduren, B. Allen, J. Cole, J. Lassiter, S. Budak, T. Colon, C. Muntele, M.A. Alim, S. Bhattacharjee, R. B. Johnson. Thermoelectric generators from SiO <sub>2</sub> /SiO <sub>2</sub> +Ge nanolayer thin films modified by MeV Si ions, Solid-State Electronics, (01 2015): 0. doi: 10.1016/j.sse.2014.08.005
04/28/2015 34.00	S. Budak, S.Guner, C. I. Muntele, D. Ila. Thermoelectric Figures of Merit of Zn <sub>4</sub> Sb <sub>3</sub> and ZrNiSn-based Half-Heusler Compounds Influenced by MeV Ion-beam Bombardments, Physics Procedia, CAARI 2014, (05 2014): 0. doi:
04/28/2015 33.00	S. Budak, M. A. Alim, S. Bhattacharjee, C. Muntele. Effects of MeV Si Ions and Thermal Annealing on Thermoelectric and Optical Properties of SiO <sub>2</sub> /SiO <sub>2</sub> +Ge Multi-Nanolayer Thin Films, Physics Procedia, CAARI 2014, (05 2014): 0. doi:
04/28/2015 32.00	C. Muntele, R. B. Johnson, S. Budak, M. Baker, J. Lassiter, C. Smith. Effects of Thermal Annealing on the Thermoelectric and Optical Properties of SiO <sub>2</sub> /SiO <sub>2</sub> +Cu Nanolayer Thin Films, Journal of Electronic Materials, (09 2014): 0. doi: 10.1007/s11664-014-3386-9
08/26/2014 23.00	K. Heidary, R.B. Johnson, T. Colon, C. Muntele, D. Ila, S. Budak. MeV Si ion modifications on the thermoelectric generators from Si/Si+Ge superlattice nano-layered films, Applied Surface Science, (02 2014): 221. doi: 10.1016/j.apsusc.2014.02.122
08/26/2014 25.00	S. Budak, S. Guner, R.A. Minamisawa, C.I. Muntele, D. Ila. Thermoelectric properties of Zn <sub>4</sub> Sb <sub>3</sub> /CeFe(4? x)CoSb <sub>12</sub> nano-layered superlattices modified by MeV Si ion beam, Applied Surface Science, (02 2014): 226. doi: 10.1016/j.apsusc.2014.04.026
08/26/2014 24.00	S. Güner, S. Budak, B. Gibson, D. Ila. Optical properties of Ag nanoclusters formed by irradiation and annealing of SiO <sub>2</sub> /SiO <sub>2</sub> :Ag thin films, Applied Surface Science, (02 2014): 180. doi: 10.1016/j.apsusc.2014.01.202
09/02/2013 12.00	S. Budak, C. Smith, C. Muntele, B. Chhay, K. Heidary, R. B. Johnson, D. Ila. Thermoelectric properties of SiO <sub>2</sub> /SiO <sub>2</sub> +CoSb multi-nanolayered thin films modified by MeV Si ions, Journal of Intelligent Material Systems and Structures, (12 2012): 1. doi: 10.1177/1045389X12470302
09/02/2013 13.00	S. Budak, R. Parker, C. Smith, C. Muntele, K. Heidary, R. B. Johnson, D. Ila. Superlattice multilayered thin films of SiO <sub>2</sub> /SiO <sub>2</sub> + Ge for thermoelectric device applications, Journal of Intelligent Material Systems and Structures, (04 2013): 1. doi: 10.1177/1045389X13483022
09/03/2013 19.00	C. Smith, M. Pugh, K. Heidary, T. Colon, R.B. Johnson, S. Budak, C. Muntele, D. Ila. MeV Si ions bombardments effects on thermoelectric properties of SiO <sub>2</sub> /SiO <sub>2</sub> +Ge nanolayers, Radiation Physics and Chemistry, (04 2012): 410. doi: 10.1016/j.radphyschem.2011.12.035
09/03/2013 18.00	S. Budaka, C. Smith, J. Chacha, C. Muntele, D. Ila. Characterization of gold nanodots arrangements in SiO <sub>2</sub> /SiO <sub>2</sub> +Au nanostructured metamaterials, Radiation Effects & Defects in Solids, (08 2012): 607. doi:

**TOTAL: 12**

Number of Papers published in peer-reviewed journals:

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**(b) Papers published in non-peer-reviewed journals (N/A for none)**

<u>Received</u>	<u>Paper</u>
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**TOTAL:**

Number of Papers published in non peer-reviewed journals:

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**(c) Presentations**

- Satilmis Budak, “Design of Nano-Structures for Energy Efficient Devices”, NanoBio Summit 2014, The University of Alabama, October 23-24, 2014 (invited talk).
- C. Bircfield, A. Tramble, C. Casselbery, C. Payton, J. Cole, S. Budak, “Effects of MeV Si ions and Thermal Annealing on Thermoelectric and Optical Properties of Multilayer Thin Films”, Tuskegee University Science and Technology Open House, Renaissance Montgomery Hotel & Spa at the Convention Center, Montgomery, AL, USA, January 30-31, 2015.
- A. Tramble, C. Casselbery, “Fabrication and Characterization of Thermoelectric Devices From Different Multilayer Thin Films”, STEM Day-2015, Alabama A&M University, Wellness Center, April 10, 2015.

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**Non Peer-Reviewed Conference Proceeding publications (other than abstracts):**

<u>Received</u>	<u>Paper</u>
08/23/2013 2.00	S. Budak, S. Kucuksari, Z. Xiao, S. Yang, B. Allen, K. Heidary, R. B. Johnson, C. Muntele . High Energy Beam Effects on the Thermoelectric and Optical Properties of SiO <sub>2</sub> /SiO <sub>2</sub> +Au Superlattice Thin Films, MRS-Fall-2013 Meeting. 01-DEC-13, . . ,
08/23/2013 11.00	B. Zheng, S. Budak, R. L. Zimmerman, D. Ila. Thermoelectric properties of MeV Si ion bombarded Bi <sub>2</sub> Te <sub>3</sub> /Sb <sub>2</sub> Te <sub>3</sub> superlattice deposited by magnetron sputtering, SMMIB-2013 Meeting. 15-SEP-13, . . ,
08/23/2013 10.00	S. Budak, B. Gibson, D. Ila, S. Günera. Optical properties of Ag nanoclusters formed by irradiation and annealing of SiO <sub>2</sub> /SiO <sub>2</sub> : Ag thin films, SMMIB-2013 Meeting. 15-SEP-13, . . ,
08/23/2013 9.00	S. Budak, S.Guner, C. I. Muntele, D. Ila. Thermoelectric Figures of Merit of Zn <sub>4</sub> Sb <sub>3</sub> and ZrNiSn-based half-heusler Compounds Influenced by MeV ion-beam bombardments, SMMIB-2013 Meeting. 15-SEP-13, . . ,
08/23/2013 8.00	S. Budaka, S.Gunerb, R. A. Minamisawac, C. I. Munteled, D. Ila. Thermoelectric Properties of Zn <sub>4</sub> Sb <sub>3</sub> /CeFe(4-x)Co <sub>x</sub> Sb <sub>12</sub> Nano-layered Superlattices Modified by MeV Si ions Beam, SMMIB-2013. 15-SEP-13, . . ,
08/23/2013 7.00	S. Budak, J. Lessiter, T. Colon, C. Muntele, S. Bhattacharjee, M. A. Alim, R. B. Johnson. Thermoelectric Generators from SiO <sub>2</sub> /SiO <sub>2</sub> +Ge Nanolayers Modified by MeV Si Ions, SMMIB-2013 Meeting. 15-SEP-13, . . ,
08/23/2013 6.00	S. Budak, M. Baker, J. Lassiter, C. Smith, R. B. Johnson . Thermoelectric and Optical Properties of SiO <sub>2</sub> /SiO <sub>2</sub> +Cu Nanolayered Thin Films Affected by Thermal Annealing , SMMIB-2013 Meeting. 15-SEP-13, . . ,
08/23/2013 5.00	S. Budak, K. Heidary, R. B. Johnson, T. Colon, C. Muntele, D. Ila. MeV Si Ion Modifications on the Thermoelectric Generator from Si/Si+Ge Superlattice Nanolayered Films , SMMIB-2013 Meeting. 15-SEP-13, . . ,
08/23/2013 4.00	S. Budak, E. Gulduren, J. Lessiter, T. Colon, C. Smith, R. Parker, C. Muntele, R. B. Johnson. Thermoelectric and Optical Properties of Si/Si+Sb Nanolayered Thin Films Effected by High Energy Beam, SMMIB-2013 Meeting. 15-SEP-13, . . ,
08/23/2013 3.00	S. Budak, S.Guner, C. Muntele, D. Ila. HIGH ENERGY IONS BEAM EFFECTS ON THE THERMOELECTRIC GENERATORS FROM AgBiTe and AgSbTe THIN FILMS, SMMIB-2013 Meeting. 15-AUG-13, . . ,
08/23/2013 1.00	S. Budak, S. Kucuksari, Z. Xiao, S. Yang, B. Allen, M. A. Alim, K. Heidary, R. B. Johnson, S. Bhattacharjee, C. Muntele. Thermoelectric and Optical Properties of SiO <sub>2</sub> /SiO <sub>2</sub> +Ge Multi-Nanolayer Thin Films Modified by MeV Si Ions , MRS-Fall-2013. 01-DEC-13, . . ,
09/02/2013 14.00	Satilmis Budak, M. Baker, C. Smith, M. A Alim, R. B. Johnson. Thickness and High Energy Beam Effects on Thermoelectric Generator of Si/Si+Ge Nanolayered Thin Films , MRS Spring 2013 (H: Nanoscale Thermoelectrics—Materials and Transport Phenomena - II). 01-APR-13, . . ,

09/02/2013 15.00 Satilmis Budak, M. Baker, C. Smith, M. A Alim, R. B. Johnson. High Energy Effects on Thermoelectric and Optical Properties of Si/Si+Sb Nanolayered Thin Films,  
MRS Spring 2013 (H: Nanoscale Thermoelectrics—Materials and Transport Phenomena - II). 01-APR-13, . : ,

09/02/2013 16.00 Satilmis Budak, M. Baker, C. Smith, M. A Alim, R. B. Johnson. Thermal Annealing Affects on the Thermoelectric and Optical Properties of SiO<sub>2</sub>/SiO<sub>2</sub>+Cu Nanolayered Thin Films,  
MRS Spring 2013 (H: Nanoscale Thermoelectrics—Materials and Transport Phenomena - II). 01-APR-13, . : ,

**TOTAL: 14**

**Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):**

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**Peer-Reviewed Conference Proceeding publications (other than abstracts):**

Received

Paper

08/26/2014 20.00 M. A. Alim, S. Bhattacharjee, C. Muntele, S. Budak. Effects of MeV Si Ions and Thermal Annealing on Thermoelectric and Optical Properties of SiO<sub>2</sub>/SiO<sub>2</sub>+Ge Multi-Nanolayer Thin Films,  
23rd Conference on Application of Accelerators in Research and Industry, CAARI 2014. 25-MAY-14, . : ,

08/26/2014 21.00 S. Budak, M. A. Alim, Z. Xiao, R. B. Johnson, S. Yang. Thermoelectric and Optical Properties of SiO<sub>2</sub>/SiO<sub>2</sub>+Au Multilayer Thin Films Affected by Thermal Annealing,  
23rd Conference on Application of Accelerators in Research and Industry, CAARI 2014. 25-MAY-14, . : ,

08/26/2014 22.00 S. Budak, S. Guner, C. I. Muntele, D. Ila. Thermoelectric Figures of Merit of Zn<sub>4</sub>Sb<sub>3</sub> and ZrNiSn-based Half-Heusler Compounds Influenced by MeV Ion-beam Bombardments,  
23rd Conference on Application of Accelerators in Research and Industry, CAARI 2014. 25-MAY-14, . : ,

**TOTAL: 3**

**Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):**

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**(d) Manuscripts**

<u>Received</u>	<u>Paper</u>
08/26/2014 26.00	Satilmis Budak, M. Baker, J. Lassiter, C. Smith, C. Muntele, R. B. Johnson. Thermal Annealing Effects on the Thermoelectric and Optical Properties of SiO <sub>2</sub> /SiO <sub>2</sub> +Cu Nanolayered Thin Films, Journal of Electronic Materials (05 2014)
08/26/2014 27.00	S. Budak, E. Gulduren, B. Allen, J. Cole, J. Lassiter, T. Colon, C. Muntele, M. A. Alim, S. Bhattacharjee, R. B. Johnson. Thermoelectric Generators from SiO <sub>2</sub> /SiO <sub>2</sub> +Ge Nanolayer Thin Films Modified by MeV Si Ions, Solid State Electronics (06 2014)
08/26/2014 29.00	Satilmis Budak, E. Gulduren, B. Allen, J. Cole, J. Lassiter, T. Colon, C. Muntele, R. Parker, C. Smith, R. B. Johnson. Thermoelectric and Optical Properties of Si/Si+Sb Multinanolayered Thin Films Effected by High Energy Radiation, Journal of Electronic Materials (07 2014)
08/26/2014 28.00	Satilmis Budak, S. Guner, C. Muntele, D. ILA. THERMOELECTRIC GENERATORS FROM AgBiTe and AgSbTe THIN FILMS MODIFIED BY HIGH ENERGY BEAM, Journal of Electronic Materials (06 2014)
08/28/2014 30.00	Satilmis Budak, Sudip Bhattacharjee, Mohammad A. Alim. The AC Electrical Behavior of Multi-Layered Thermoelectric Devices, Physica Status Solidi A: Applications and Materials Science (07 2014)
<b>TOTAL:</b>	<b>5</b>

**Number of Manuscripts:**

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**Books**

<u>Received</u>	<u>Book</u>
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**TOTAL:**



Received

Book Chapter

**TOTAL:**

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**Patents Submitted**

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**Patents Awarded**

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**Awards**

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**Graduate Students**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Brandon Allen	0.00	
Jorden Cole	0.00	
<b>FTE Equivalent:</b>	<b>0.00</b>	
<b>Total Number:</b>	<b>2</b>	

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**Names of Post Doctorates**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
<b>FTE Equivalent:</b>	
<b>Total Number:</b>	

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**Names of Faculty Supported**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Dr. S. Budak	0.04	
Dr. M. Alim	0.00	
Dr. R. B. Johnson	0.00	
Dr. Z. Xiao	0.00	
<b>FTE Equivalent:</b>	<b>0.04</b>	
<b>Total Number:</b>	<b>4</b>	

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### Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Aomzada McCaslin	0.00	Electrical Engineering
Nhan Mai	0.00	Electrical Engineering
Charles Payton	0.00	Physics
Zack Duncan	0.00	Electrical Engineering
Ashley Tramble	0.00	Electrical Engineering
Chauncy Casselbery	0.00	Electrical Engineering
Cody Birchfield	0.00	Electrical Engineering
<b>FTE Equivalent:</b>	<b>0.00</b>	
<b>Total Number:</b>	<b>7</b>	

### Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: ..... 1.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 1.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 1.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense ..... 1.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:..... 0.00

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### Names of Personnel receiving masters degrees

<u>NAME</u>
<b>Total Number:</b>

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### Names of personnel receiving PHDs

<u>NAME</u>
<b>Total Number:</b>

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### Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
<b>FTE Equivalent:</b>	
<b>Total Number:</b>	

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### Sub Contractors (DD882)

**Inventions (DD882)**

**Scientific Progress**

**Technology Transfer**

## INTRODUCTION

The research objective of this project, led by Principal Investigator Dr. Satilmis Budak, at Alabama A&M University (AAMU), a leading HBCU, was to use nano-engineering and nanofabrication to develop nanostructured thermoelectric (TE) materials for application in high-efficiency thermoelectric power generators and solid-state micro cooling devices. This report is the Final Report for the Project and also summarizes the research activities accomplished during the short period following our third interim report. A variety of multi-nano-layered superlattice thin films, based on  $\text{Bi}_2\text{Te}_3/\text{Sb}_2\text{Te}_3$ ,  $\text{SiO}_2/\text{SiO}_2+\text{CoSb}$ ,  $\text{SiO}_2/\text{SiO}_2+\text{Au}$ ,  $\text{Zn}_4\text{Sb}_3/\text{CeFe}_{(4-x)}\text{Co}_x\text{Sb}_{12}$ ,  $\text{SiO}_2/\text{SiO}_2+\text{Ge}$ ,  $\text{SiO}_2/\text{SiO}_2+\text{Cu}$ ,  $\text{AgBiTe}$ ,  $\text{AgSbTe}$ ,  $\text{Si/Si+Sb}$ ,  $\text{Si/Si+Ge}$ ,  $\text{Zn}_4\text{Sb}_3$ , and  $\text{ZrNiSn}$ , were grown using ultra-high vacuum-based deposition methods. Figure 1 illustrates a typical multilayer thin-film system. These thin films were further modified by MeV Si-ion beam bombardment to produce nanodots and nanoclusters for achieving high thermoelectric figure of merit ( $ZT$ ). To the best of our knowledge, our research team is the only group using high-energy ion-beam technology to enhance the thermoelectric figure-of-merit. The complexity of fabrication of these thin-film systems is significant and is thoroughly discussed in the numerous papers and poster presentations our team has produced and given. The MeV Si-ion beam bombardment requires specialized equipment that AAMU has on campus and significant operator experience. The principal anticipated outcome of our research was to significantly increase the  $ZT$  value by increasing both the Seebeck coefficient and electrical conductivity while decreasing thermal conductivity. By doing so, it is expected that nano-power thermoelectric generators for heat harvesting and thermoelectric coolers for achieving cryogenic temperatures can be realized. AAMU asserts that the Project was successful in achieving the Project objectives and, as reported in the literature, our team arguably holds the world record for room-temperature  $ZT$  with a value of almost five (5). Additional research should be supported to bring the technology that AAMU has developed from TRL 3 to TRL 4–5. During the conduct of this Project, a total of 12 peer-reviewed papers and 3 peer-reviewed conference proceedings were published, 14 non-peer reviewed conference proceeding and poster presentations were given, 15 presentation were presented, and 28 undergraduate students and 4 graduate students were involved earning 10 B.S. Degrees and 2 M.S. Degrees.

Single Layer	<b>Au</b>	100nm
Multilayer	<b>Ge/Si+Ge</b>	200nm
Single Layer	<b>Si +Ge</b>	100nm
Single Layer	<b>Si</b>	100nm
Single Layer	<b>Sb+Ge</b>	50 nm
Single Layer	<b>Ge</b>	60 nm
<b>Multilayer</b>	<b>SiO<sub>2</sub>/SiO<sub>2</sub> + Ge</b>	12nm
Single Layer	<b>Au</b>	100nm
	<b>Substrate</b>	

Figure 1: Cross-section of geometry used for typical multilayer thin-film system showing combination of single layers and multilayers.

During the first reporting period, beginning February 01 and ending July 31, 2012, several measurement systems were ordered. An Agilent cross-plane electrical conductivity measurement system was ordered and received at the laboratory during this initial period. The MMR Technology Seebeck and Van der Pauw - Hall Effect measurement system and the Filmetrics UV-Region high sensitivity thin-film thickness measurement system were received during the reporting period beginning August 1, 2012 and ending July 31, 2013. All three instruments were installed and made operational, and have been great assets in this research effort. In 2013, the PI and Co-PIs submitted an instrumentation grant proposal to the NSF-MRI program to purchase a high-sensitivity thermal-conductivity measurement system. The grant was awarded and the ULVAC-Laser PIT Diffusivity system was ordered (\$189,300) and the equipment was received by the Department of Electrical Engineering and Computer Science. The PI and Co-PIs were trained on how to use the Laser PIT system. This system complements our 3<sup>rd</sup> Harmonic system and has further enhanced the investigative capability of the subject research Project.

## **RESEARCH ACTIVITIES** (August 1, 2014 – January, 31, 2015)

During the final period of this Project, the following activities occurred.

- Two Electrical Engineering Senior Design students worked on the Project;

- Two Master's Degree students in the Materiel Engineering M.S. Program of the Department of Electrical Engineering and Computer Science continued their research (Jorden Cole, Branden Allen);
- One undergraduate student from the Department of Electrical Engineering worked on the Project under the support of the NSF-EPSCOR program during August 15, 2014-April 30 2015;
- Four undergraduate students from the Department of Electrical Engineering worked on the Project under the support of DOE-NNSA program during August 15, 2014-April 30 2015; and
- Dr. Claudiu Muntele helped on the high-energy ion bombardment and RBS measurements of the fabricated multi and single-layered thin film systems.

The last period of the Project was very productive with the publication of five papers and three presentations which their citations follow.

#### **Published Journal Articles:**

- S. Budak, S. Guner, C. Muntele, D. Ila, "Thermoelectric Generators form AgBiTe and AgSbTe Thin films Modified by High Energy Beam", Journal of Electronic Materials (DOI 10.1007/s11664-014-3581-8, online published: 08 January 2015).
- S. Budak, E. Gulduren, B. Allen, J. Cole, J. Lassiter, T. Colon, C. Muntele, M. A. Alim, S. Bhattacharjee, R. B. Johnson, "Thermoelectric Generators from SiO<sub>2</sub>/SiO<sub>2</sub>+Ge Nanolayers Modified by MeV Si Ions", Solid-State Electronics 103 (2015) 131–139.
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- S. Budak, M. Baker, J. Lassiter, C. Smith, C. Muntele, R. B. Johnson, “Thermoelectric and Optical Properties of SiO<sub>2</sub>/SiO<sub>2</sub>+Cu Nanolayered Thin Films Affected by Thermal Annealing”, Journal of Electronic Materials, doi: 10.1007/s11664-014-3386-9 (2014-The Minerals, Metals & Materials Society).

#### **2014 Presentations:**

- A. Tramble, C. Casselbery, “Fabrication and Characterization of Thermoelectric Devices From Different Multilayer Thin Films”, STEM Day-2015, Alabama A&M University, Wellness Center, April 10, 2015.
- C. Bircfield, A. Tramble, C. Casselbery, C. Payton, J. Cole, S. Budak, “Effects of MeV Si ions and Thermal Annealing on Thermoelectric and Optical Properties of Multilayer Thin Films”, Tuskegee University Science and Technology Open House, Renaissance Montgomery Hotel & Spa at the Convention Center, Montgomery, AL, USA, January 30-31, 2015.
- Satilmis Budak, “Design of Nano-Structures for Energy Efficient Devices”, NanoBio Summit 2014, The University of Alabama, October 23-24, 2014 (invited talk).

#### **Specific Activities:**

Prepared thermoelectric devices were bombarded with 5 MeV Si ions at the different fluences (doses) to form quantum dots (nano dots) in the multilayers to improve the efficiency of the thermoelectric devices. The Seebeck coefficients of the prepared devices for the different multilayer thin film systems were measured. Figure 2 shows the Seebeck coefficient of the thin films at the different fluences under the different temperatures.

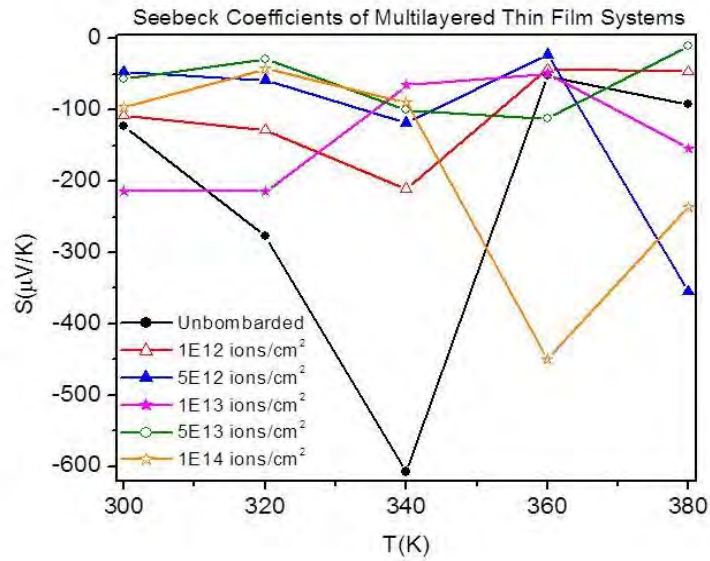


Figure 2. Seebeck Coefficients of Multilayered Thin Films

After obtaining the Seebeck measurements, the students performed van der Pauw four-probe resistivity, Hall Effect, density, mobility measurements as shown in Figures 4–6 below:

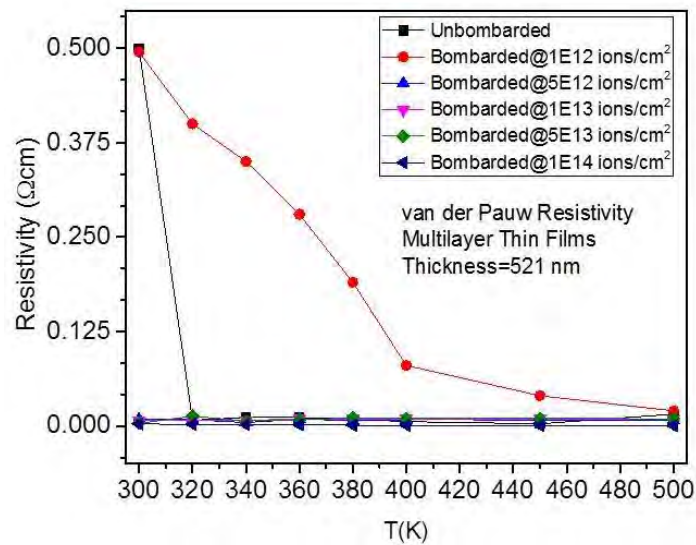


Figure 3. Van der Pauw resistivity measurements of multilayered thin films.



The resistivity measurement in Figure 3 provides the reciprocal of the electrical conductivity needed for efficiency measurement. They also recorded other data from van der Pauw including density, mobility and Hall coefficient. Figures 4 through 6 show these results.

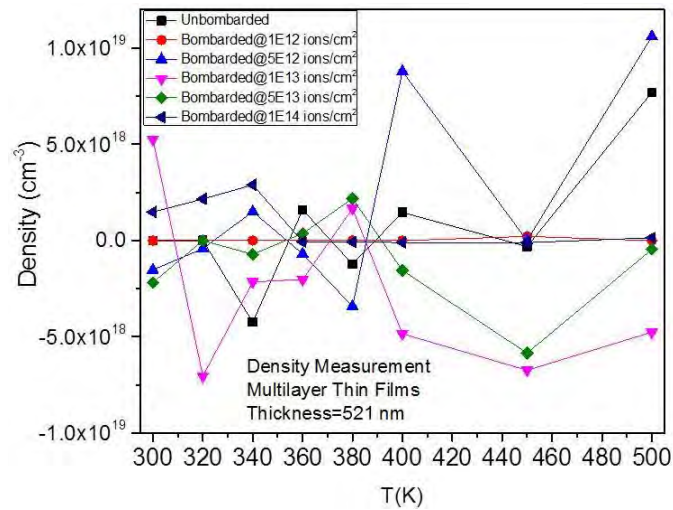


Figure 4. Van der Pauw density measurement of multilayered thin films.

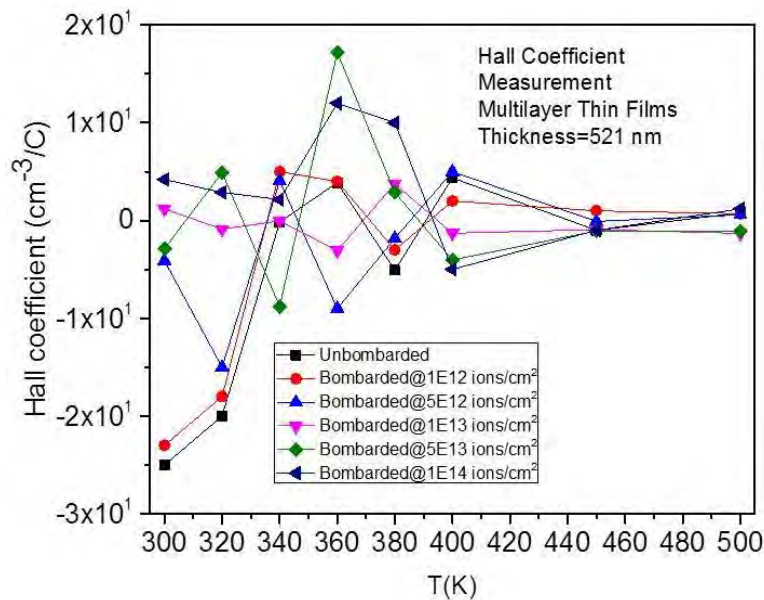


Figure 5. Van der Pauw Hall coefficient measurement of multilayered thin films.

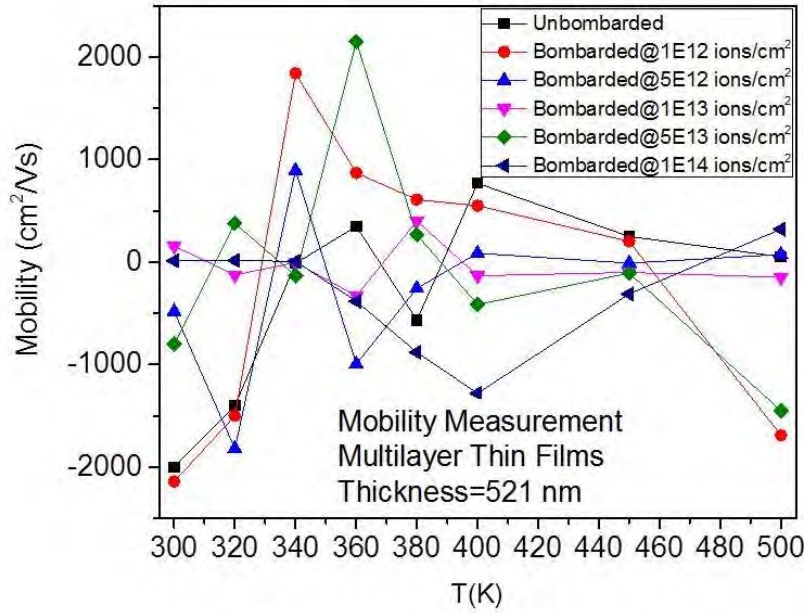


Figure 6. Van der Pauw mobility measurements of multilayered thin films.

Students measured the thickness of the 22 different multilayer coated devices. These cut wafers were in the chamber with our device. Our devices were masked for the deposition and these measured samples were not. The samples were completely covered in our multilayer and were used for thickness measurement of the multilayer. The samples measured as follows:

A)521.9nm	E)520.9 nm	I)521.7 nm	M)521.4nm	Q)521.2nm	U)521.2nm
B)521.4nm	F) 522nm	J)521.3 nm	N)521.8nm	R)521.4 nm	V)521nm
C)521.6nm	G)521.2nm	K)521 nm	O)521.7nm	S)521.8nm	
D)521.2nm	H)521.5nm	L)521.5 nm	P)520.7nm	T)521.1 nm	

The average thickness computed to be 521.3864 nm with a standard deviation of 0.344059041 nm. After thickness measurements were completed, images of the devices were taken by the students using our Scanning Electron Microscope (SEM) and Energy-dispersive X-ray Spectroscopy (EDS). Figure 7 shows the surface of our device using the SEM and demonstrates the desired smooth surface. Figures 8 and 9 show results obtained from the EDS while Figures 10 and 11 show a presentation and students and faculty at work in the laboratory.

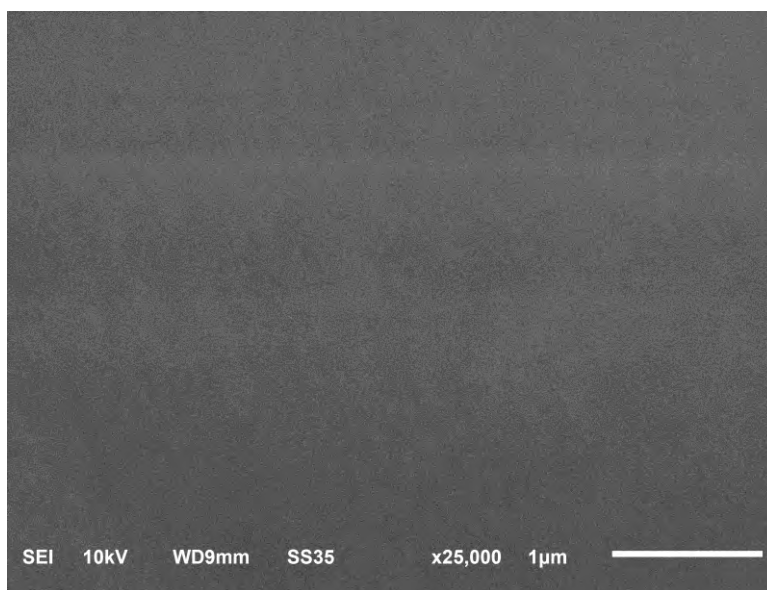


Figure 7. Picture from SEM

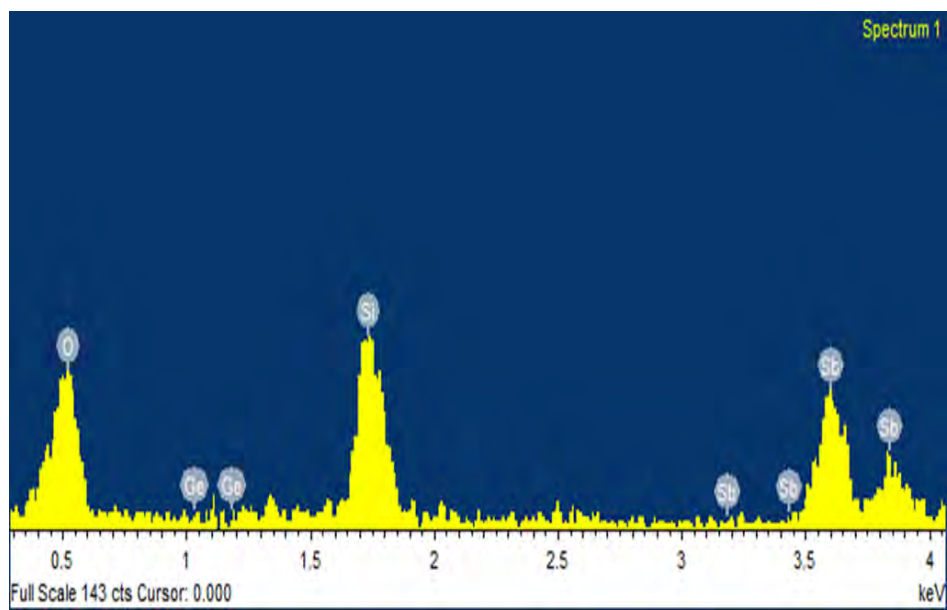


Figure 8. Picture from EDS

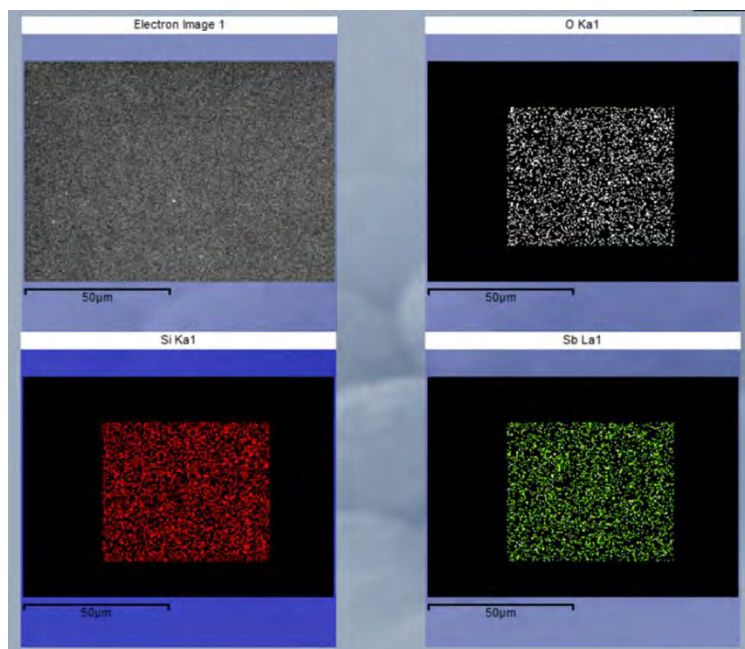


Figure 9. Picture of separated elements from EDS (elemental mapping of used materials at the surface of the fabricated devices).

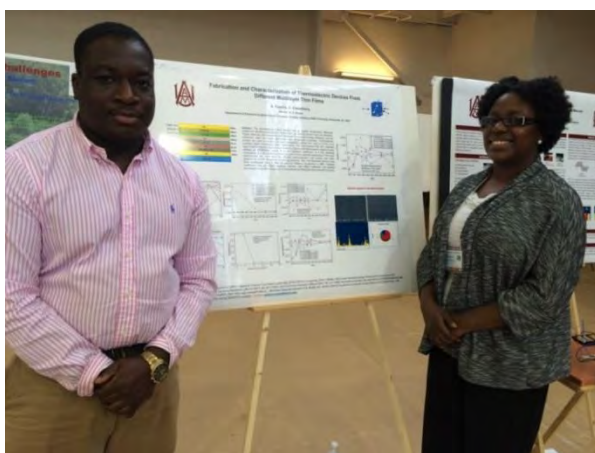


Figure 10. A. Tramble, C. Casselbery, “Fabrication and Characterization of Thermoelectric Devices From Different Multilayer Thin Films”, STEM Day-2015, Alabama A&M University, Wellness Center, April 10, 2015.





Figure 11. Students were working in Clean Room.

## SUMMARY

This three-year Project gave us extensive experience and the achievement of remarkable results not addressed in the literature before our publications. Currently, we have attained the  $ZT$  figure-of-merit of 4.0 and 4.9 from the 100 multilayers of  $\text{SiO}_2/\text{SiO}_2+\text{Ge}$  thin films reported in our previous interim reports. These values are the highest values for the multi-nanolayered thin-film systems and the highest reported room-temperature  $ZT$  in the literature. A number of students gained very valuable laboratory and research experience that will help them in their career. They also gained a practical understanding of practicing the scientific method. AAMU would like to continue, given future research funding, with our new design for the thermoelectric and photovoltaic systems and hybrid thermoelectric-PV systems which could work at significantly high temperatures. We are confident that our team can work on the different multilayered thin film systems combined together and utilizing additional thermos-tunneling methods to realize even the higher efficiencies ( $ZT$ ).